07-Crystallography of Organometallic and Coordination Compounds

a=9.358(1), b=13.408(1), c=13.755(5) Å, space group: Pnma, R=0.042 for 1590 reflections. Those of the second compound are: monoclinic, a=9.688(2), b=14.157(2), c=25.520(3), \( \beta = 94.72(1) ^\circ \), space group: P2_1/n, R=0.044 for 3297 reflections. Those of the third compound are: monoclinic, a=8.050(1), b=12.490(2), c=20.193(4), \( \beta = 95.97(1) ^\circ \), space group: P2_1/c, R=0.024 for 3542 reflections. [Work was supported by National Science Council, Taiwan, China]

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PS-97.04.44 THE 2PREPARATION AND CRYSTAL STRUCTURE OF (CuHgNH₃)₂SnC₅. By Wei Wang, Yonghua Lin, Laiming Li and Shiquan Xi, Changzhen Institute of Applied Chemistry, Chinese Academy of Sciences, Changzhen 100022, P. R. China.

The bis(n-decylammonium) tetrahelometallates(II) are known to crystallize in a perovskite-type bidimensional structure. They are of much current interest from both magnetic and structural points of view (K. J. Schenk, G. Chapuis, J. Phys. Chem., 1988, 92, 741). But very few studies have been performed on bis(n-alkylammonium) hexahelometallates(IV) with general formula (n-C₆H₁₃NH₃)xSnC₅. Up to now, no structure of a long-chain bis(n-alkylammonium) hexahelometallates(IV) has been reported. We have prepared (C₁₂H₂₅NH₃)xSnC₅ (abbreviated as C₁₂Sn) and determined its crystal structure.

The colorless plate-shaped crystals of C₁₂Sn were grown at room temperature from absolute alcohol solution containing decylammonium chloride and SnCl₄. Intensity data were collected using a Nicolet RSM/E diffractometer. The structure was solved by the Patterson method and final R=0.069 for 2148 unique reflections [1=3a (0)]. At room temperature the crystal is monoclinic with a=11.900(4)Å, b=7.280(3)Å, c=9.600(1)Å, \( \beta = 94.08(3)^\circ \), \( \lambda = 0.00630(20) \)Å, and belongs to the space group P2₁/c, with four molecules in the unit cell. The structure of C₁₂Sn is characterized by a layer of almost regular SnCl₄- octahedra sandwiched between two hydrocarbon layers. The NH₃ polar heads of the decylammonium cations are linked to the chloride atoms by three N...Cl hydrogen bonds. There are two types of inequivalent hydrocarbon chains which are packed together. One has a perfectly ordered all-trans conformation, and the other has an extended conformation with only a single gauche turn between the second and the third carbon atoms. The general arrangement of the alkyl chain of C₁₂Sn is comparable to the bilayer structure of biological membranes.

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PS-97.04.45 THE CRYSTAL AND MOLECULAR STRUCTURE OF [1,2-DIMETHYL-1,2-DI(2-ISOBUTYLETHYL)BICYCLOPENTADIENYL TITANIUM DICHLORIDE. By Zhi-tao Wang, Shou-shan Chen, Ru-ji Wang and Xin-han Yao, Central Laboratory, Institute of Elemento-organic Chemistry, Nankai University, Tianjin 300071, China.

The study of chiral bridged cyclopentadienylmetal complexes has now become a very active field in organometallic chemistry. We report the structure determination of a new compound C₁₂H₂₅C₅Ti by X-ray crystallography.

A sample was recrystallized from mixed solvents of dichloromethane and petroleum ether and transparent crystals, intensities were collected on a CAD4 diffractometer, \( \omega-2 \theta \) scan mode, MoK\( _{\alpha} \) in the range of 2.0 < 20 < 25.0. 2000 independent reflections were measured, of which 1344 were observed reflections with \( 1 < 2 \theta < 3 \mathrm{H} \). The intensities were corrected for Lorentz and polarization.

This compound crystallizes in the monoclinic space group C 2/c with unit cell parameters: a=13.217(3)Å, b=9.496(2)Å, c=16.449(8)Å, \( \beta = 94.75(1)^\circ \), \( \chi = 2057.3 \AA^2 \), \( M_r = 415.31 \), \( Z = 4 \), \( D_x = 1.34 \mathrm{g/cm}^3 \), \( \mu = 6.70 \mathrm{cm}^{-1} \), F(000) = 880.

The structure was solved by direct method (MALTAN-82) and subsequent difference Fourier syntheses. Full-matrix least-squares refinement with anisotropic thermal parameters for non-hydrogen atoms led to an R of 0.065 and an Rw of 0.070.

The molecule is shown in Figure 1. Its molecular structure possesses C₂ symmetry which belongs to the type of equivalent homotopic faces of cyclopentadienyl ligands (Ronald L. Halterman, Chem. Rev., 1992, 92, 965-994). There is a half molecule in an asymmetric unit, the second half is generated by C₂ symmetry, the dihedral angle between two cyclopentadienyl planes is 63.35(5)°.